U.S Environmental Protection Agency
Office of Research and Development
National Exposure Research Laboratory
Exposure Methods and Measurements Division (EMMD)
Air Quality Branch (AQB)

STANDARD OPERATING PROCEDURE

SOP Title: Operation of the Aerodyne/Tofwerk Chemical Ionization Mass Spectrometer

SOP ID: D-EMMD-AQB-SOP-3067-1	Effective Date: December 15, 2017

SOP was Developed: ☒ In-house ☐ Extramural:

SOP Discipline*: General Chemistry

Alternative Identification: Previous EMMD ID#: D-EMMD-AQB-013-SOP-02

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Revision History

Version No.	Name	Date of Revision	Description of Change(s)
1	Theran Riedel	01/01/2016	Original document created EMMD ID# D-EMMD-AQB-013-SOP-01 QA Track ID: D-EMMD-AQB-SOP-3067-0
2	Theran Riedel	12/15/2017	Added new instrument and software items EMMD ID# D-EMMD-AQB-013-SOP-02 QA Track ID: D-EMMD-AQB-SOP-3067-1

Table of Contents

1. Scope and Application

This Standard Operating Procedure/Recommended Operating Procedure (SOP/ROP) applies specifically to the use of the Aerodyne/Tofwerk high resolution time-of-flight chemical ionization mass spectrometer (CIMS – also referred to as the ToF-CIMS) operated in the negative mode for measurements of gas-phase species. Applicable reagent ions for this procedure include, but are not limited to, iodide and acetate. This should serve as a general reference for experienced users only. New users should be thoroughly trained by experienced users prior to independent operation.

2. Summary of Method

The following establishes general operating procedures for obtaining gas-phase mass spectra and also describes how to appropriately vent and pump down the CIMS.

3. Definitions

ADC:	Analog-to-Digital Converter
AMS:	Aerodyne Aerosol Mass Spectrometer
CIMS:	high resolution time-of-flight Chemical Ionization Mass Spectrometer
cps:	counts per second
DC:	Direct Current
IMR:	Ion-Molecule Region
LN2:	liquid nitrogen
m/z:	mass-to-charge ratio
MCP:	Micro Channel Plate detector
MFC:	Mass Flow Controller
MS:	Mass Spectra
MSDS:	Material Safety Data Sheets
N_2:	nitrogen gas
PTRMS:	Proton Transfer Reaction Mass Spectrometer
slpm:	Standard Liters Per Minute
SOP:	Standard Operating Procedure
SSQ:	Short Segmented Quadrupole
TIC:	Total Ion Current
TPS:	ToF Power Supply

4. Health and Safety Warnings

The CIMS utilizes DC voltages in excess of 1 kV for ion focusing and detection. Reagent ion source compounds are often toxic and/or severe irritants. Care must be taken to ensure that all pump exhaust is routed to a fume hood or a well ventilated outdoor location. The ion source utilizes radioactive (alpha emission) polonium-210. Unless contained in a sealed vessel, reagent ion compounds and the ionizer should be handled within a fume hood using protective gloves, lab coat, and safety glasses. Consult appropriate Material Safety Data Sheets (MSDS) prior to use. Exercise caution when handling the high-pressure liquid nitrogen (LN2) dewar that provides N_2 for CIMS operation.

5. Cautions

Operation must be carried out in a relatively clean environment.

6. Personnel Qualifications

A comprehensive working knowledge of CIMS measurements or similar techniques (Proton Transfer Reaction Mass Spectrometer (PTRMS), Aerosol Mass Spectrometer (AMS), etc.) is required to operate the CIMS and perform the associated measurements. Personnel must also have a thorough understanding of chemical ionization-mass spectrometry theory, sampling setup, high-vacuum systems, DC electronics, computer data analysis software (Igor Pro and/or MATLAB), and general instrument troubleshooting. If the topics covered hereafter are unfamiliar to you, and/or you are unable to perform the tasks confidently and independently, additional training and supervision is required before using this instrument. All training should documented using the "ORD Certification Statement for Demonstration of Capability" form provided at the end of this SOP document.

7. Equipment and Supplies

Select Equipment:

- Aerodyne/Tofwerk Time of Flight Chemical Ionization Mass Spectrometer
- CIMS PC with Acqiris AP240 and peripherals
- ToF Power Supply (TPS) (version 1)
- APC Uninterruptible Power Supply (UPS)
- Vacuubrand MD1 diaphragm pump
- Agilent IDP3 Dry scroll vacuum pump
- Agilent Triscroll 600 dry scroll pump
- Pfeiffer split flow turbo pump
- AALBORG MFC GFC

Supplies:

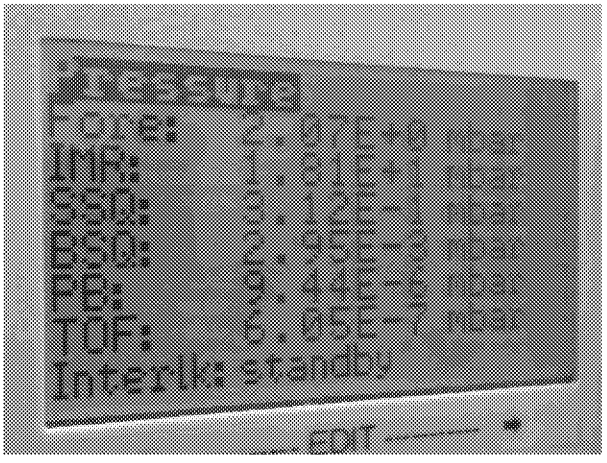
- LN2 high-pressure dewar
- NRD Static Master In-line Alpha Ionizer model P-2021
- Small flathead screw driver (attached to CIMS rack)

8. Procedure

NOTE: The official ToF-CIMS Manual and additional details regarding instrument operation are available at https://sites.google.com/site/citofms/. This site is the primary source of information related to the CIMS. Email cacc-support@aerodyne.com to request full access. This SOP/ROP should be considered a complementary source of information and not a replacement for the information available on the website. A hard copy of this manual is also contained in the CIMS binder that is always with the instrument.

A summary for a generalized procedure to perform CIMS measurements is outlined below. It is assumed that the CIMS is under vacuum, at base pressure (see Figure 8.1 – pressures within 50% of those shown in Figure 8.1 are acceptable) with all pumps running, all pump valves are completely open, and the LN2 dewar is connected to the ionizer/Ion-Molecule Region (IMR) via the mass flow controller (MFC) and reagent ion source. If any of the "mbar" labels in Figure 8.1 instead read "INTERLOCK", the system is currently interlocked and not at base pressure. If the instrument is not at base pressure, users should follow section *8.7 Pumping Down* prior to any other sections.

Figure 8.1 Typical base pressures read by the Giraffe display



If any of the steps outlined below cannot be completed, STOP. The CIMS is not in working condition. Contact a more experienced operator or cacc-support@aerodyne.com if you are unable to diagnose the problem yourself.

8.1 Giraffe controller operation

The Giraffe controller (Figure 8.2), or simply "the Giraffe", allows the operator to configure and monitor various modules that control and monitor various CIMS components. These components are grouped into submenus and include Turbo pumps, Pumps MD1 and IDP3, system Pressures, and the Heater.

General Control Buttons:

Vertical Buttons (to the left of the display screen)

- Scroll the cursor up and down to move between menu items.
- Pressed together to confirm a choice (send the command).

Horizontal Buttons (below the display screen)

- When the menu title is highlighted: scroll through the top level menus.
- When a sub-menu is highlighted: Scroll through the values of that parameter.
- Pressed together: Move into and also out of a setup menu (e.g. press the 2 buttons in the IDP3 menu to go to a menu where you can turn it on).

Vertical + Horizontal Buttons

- Pressing all four buttons together resets the Giraffe display. This is sometimes necessary if, for whatever reason, the Giraffe module does not find all device modules at start-up.

Rotating knob

- Adjust values (e.g. on-off).

Specialized Buttons:

Turbo On/Off

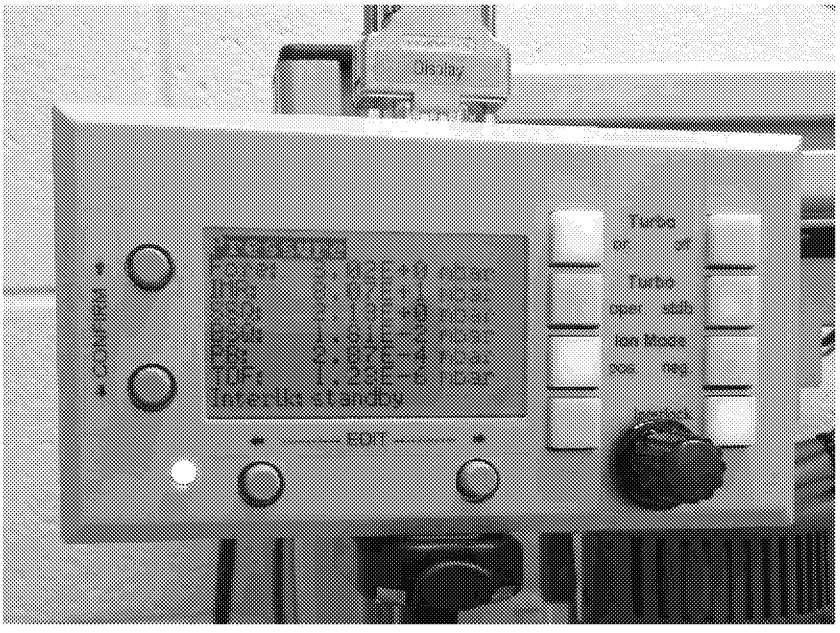
- Toggles turbo pump on/off.

Turbo Oper/Stdb

- Sets speed of turbo pump.

All other Giraffe buttons will remain unused.

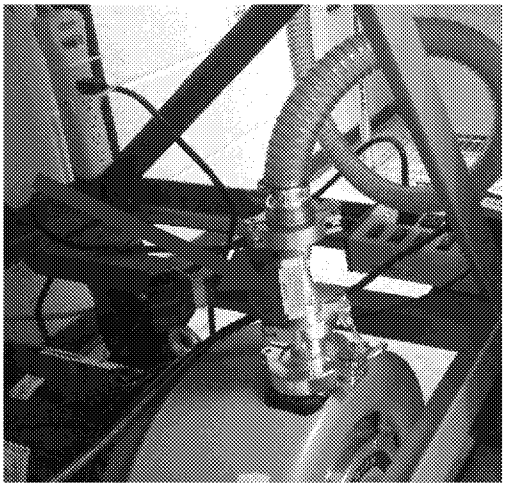
Figure 8.2 Giraffe controller



8.2 Setting the pressures

8.2.1 The Triscroll dry scroll pump equipped with a vacuum pump isolation (VPI) valve that automatically closes when power to the Triscroll is interrupted. If the Triscroll is running, the VPI valve is completely open (see Figure 8.3 for VPI valve location).

Figure 8.3 Triscroll VPI valve location



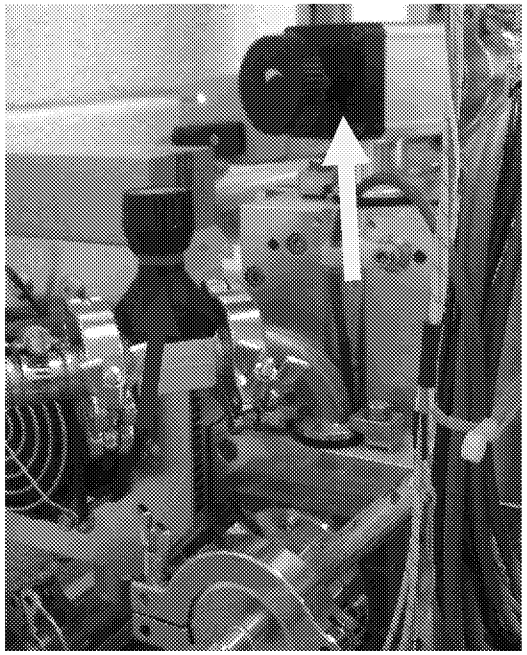
8.2.2 Remove inlet cap/plug from the CIMS inlet and connect sampling line.

8.2.3 Increase MFC flow to 1.5 slpm using a small flathead screwdriver to turn the potentiometer clockwise (see Figure 8.4)

Figure 8.4 MFC flow adjustment

8.2.4 Slowly close IMR and Short Segmented Quadrupole (SSQ) pumping valves by turning the respective knobs clockwise until IMR pressure is 100 mbar, and SSQ pressure is 2 mbar (see Figure 8.5). Pressures are monitored on the Giraffe display. When pressures are set, the resulting TOF pressure should be ~1.3e-6 mbar as displayed on the Giraffe.

Figure 8.5 IMR (red arrow) and SSQ (yellow arrow) pumping valve locations



8.3 Configuring the electronics

NOTE: The following references the getting started sections of the CIMS manual available on the CIMS user website.

8.3.1 Turn on the ToF power supply (TPS) (green switch) and ensure all LED indicator lights are green.

8.3.2 Open TPS Controller software by double clicking on the TPS Controller icon on the desktop.

8.3.3 Within the TPS Controller window, if ion mode is positive, click IM Shutdown, switch to negative polarity, and click Initialize.

8.3.4 Load appropriate *.set file (File → Load Settings), click Set All Voltages, and check to ensure voltages match set points.

8.3.5 Open TofDaq software by double clicking on the TofDaq icon on the desktop and click the acquire data (green arrow) button to begin data acquisition.

8.3.6 Confirm normal spectra with appropriate peak at reagent ion m/z and total ion current (TIC) (>1e5 cps) then stop acquisition by clicking the red square button.

8.3.7 Set the Analog-to-Digital Converter (ADC) baseline as instructed in "5.1 Setting ADC Baseline" of the manual/CIMS user website.

8.3.8 Set the ADC threshold as instructed in "5.2 Setting ADC Threshold" of the manual.

8.3.9 Measure and set the single ion area as instructed in "Using SingleIon.exe" document contained in the CIMS manual binder and described below.

8.3.9.1 Collect a MS in TofDAQ to confirm normal spectra
8.3.9.2 Open SingleIon.exe (shortcut available on the Windows Desktop)
8.3.9.3 Select the "Dynamic" check box
8.3.9.4 Press start and run until the SIS value has stabilized (~2 minutes) then press top. The new single ion area value will be automatically written and saved into the DAQ settings.

8.3.10 Click the green arrow button in the TofDaq Viewer to collect at least one mass spectrum and then click the red square button in TofDaq Viewer to stop data acquisition.

8.3.11 Click on the DAQ button in the TofDaq Viewer. On the Mass Calibration tab, click Refine and then Calibrate.

8.4 Collecting data

8.4.1 After performing sections 8.2 and 8.3 above, click the green arrow button in the TofDaq Viewer to start normal data acquisition.

8.5 Stopping data collection and returning to standby

8.5.1 Click the red square button in TofDaq Viewer to stop data acquisition.

8.5.2 Click Shutdown in TPS Controller and wait for "TPS is shutdown" message.

8.5.3 Turn off the TPS (green switch).

8.5.4 Completely open IMR and SSQ pumping valves by turning the valve knobs counterclockwise and plug inlet with an appropriate Swagelok fitting.

8.5.5 To conserve N_2, the MFC flowrate can be lowered to ~0.25 slpm by using a small flathead screwdriver to turn the potentiometer counterclockwise until the desired flowrate is reached (see Figure 8.4).

8.6 Venting
Occasionally it is necessary bring the CIMS to atmospheric pressure in order to perform maintenance items such as those listed in section 9 *General Maintenance.*

CAUTION: NEVER turn on the TPS when the system is vented. This avoids potential electrical arcing within the instrument.

8.6.1 Ensure the inlet is capped/plugged, the TPS is OFF, all pumps are active, the IMR and SSQ pumping valves are completely open, and the CIMS is at base pressure.

8.6.2 Quickly disconnect and plug/cap the MFC Swagelok fitting leading to the reagent ion source and ionizer.

8.6.3 Close the IMR and SSQ pumping valves and the Triscroll valve by rotating the valve knobs clockwise.

8.6.4 Turn off the turbo pump using the Giraffe.

8.6.5 Monitor the turbo pump frequency with the Giraffe until 0 Hz (typically ~30 minutes).

8.6.6 Turn off the MD1 pump using the Giraffe controls (under the pumping submenu). The system is now vented.

8.6.7 Optional: IDP3 pump can be turned off (via Giraffe) and Triscroll pump can be unplugged.

8.7 Pumping down
After venting the CIMS should be pumped down to base pressures as soon as possible.

8.7.1 Ensure the inlet and flow through the ionizer is plugged.

8.7.2 Turn on the MD1 and set power to 50% using Giraffe (under the pumping submenu).

8.7.3 If off, turn on the IDP3 pump (set to 90% via Giraffe) and plug in the Triscroll pump. It is normal for the Triscroll to be loud.

8.7.4 Open Triscroll, IMR, and SSQ valves completely by turning the valve knobs counterclockwise.

8.7.5 Wait until Fore, IMR, and SSQ pressures are not interlocked (i.e., "INTERLOCK" is not showing on the Giraffe display), which usually takes ~15 minutes.

8.7.6 Turn on the turbo using Giraffe. For faster pumping, use Giraffe to switch turbo to "Oper" instead of "Stdby".

8.7.7 Allow at least 3 hours of pumping to achieve base pressure in TOF region (see Figure 8.1).

8.7.8 Quickly connect MFC to reagent ion source via the Swagelok fitting and confirm base pressures. The CIMS is now pumped down.

9. General Maintenance

Very little maintenance is required to keep the CIMS in good operating condition. The system should always be kept under vacuum at base pressures, even during long-term inactivity. Some inevitable maintenance tasks are presented in Table 9.1. Instructions for performing these items are available on the CIMS user website and within the manufacturer provided pump manuals. These tasks should only be performed by or under the supervision of an experienced CIMS or other Aerodyne ToF-MS instrument (e.g., the Aerosol Mass Spectrometer) user.

Table 9.1 CIMS Maintenance Tasks and Schedule

Maintenance Procedure	Frequency	Symptoms/Comments
Micro Channel Plate (MCP) detector replacement	As needed (likely ~2 years)	MCP >2450V and no MS
Ionizer replacement	Every 12 months	P-2021 lease has expired
IDP3 pump tip seal replacement	As needed	Poor pump performance, IMR unable to achieve operating pressures
Triscroll pump tip seal replacement	As needed	Poor pump performance, SSQ unable to achieve operating pressures
Reagent ion solution replacement	As needed	Empty solution reservoir, discolored permeation tube

10. Data and Records Management

Raw data (*.h5 files) that are saved on the CIMS PC hard drive automatically during data acquisition should be backed up to an external drive as often as possible to ensure storage redundancy. Storage capacity of the CIMS PC hard drive and the backup drive should be monitored to ensure sufficient storage space. All CIMS activities including maintenance and all research activities should be recorded in the user's lab notebook.

11. Quality Control/Quality Assurance

If the above guidelines and those presented on the CIMS user website are followed, mass spectra obtained by the CIMS should be robust and reproducible when sampling an unchanging air mass. Operators attempting to quantify specific chemical compounds with this system will need to develop appropriate calibration methods and are encouraged to submit an SOP or QAPP for detailing such calibrations.

12. References

Aerodyne ToF-CIMS user website: https://sites.google.com/site/citofms/

ORD Certification Statement for Demonstration of Capability

Date: _____

Technical Instructor(s): _____

_____

Trainee(s): _____

_____

Training Topic/Technique/SOP(s)/OP(s)

Criteria Used for Determining Success:

Comments/Summary:

_____

Lead Technical Instructor Signature Date

NOTE: This certification, along with any data sheets generated in the training, is to be kept with the project study file.